Absolute Specular Reflectance Measurements at Angles and Beyond

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– What We Will be Going Over –

What are My Choices in Accessories?
Are there Advantages/Disadvantages I Need to Know?
What Kind of Measurements Can I Make?
Are there any Limitations?
How Can I Make Absolute Specular Reflectance Measurements at the Angle of My Choosing?
Examples of Sample Measurements

Wrap Up and Question Time
Thank the People That Got Us Here

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How has it Been Done in the Past?

Fixed Angle

Relative Reflectance

Absolute V-N V-W

Variable Angle
UV-Vis NIR Spectroscopy Solutions
The Agilent Cary Family

"A word from our sponsors"
Measurement Modes

1. Absolute specular reflection
2. Diffuse scattering
3. Glossy scattering
4. Direct transmission
5. Scattered transmission
6. Partial scattered transmission
Relative Reflectance Measurement

Reference Measurement

Sample Measurement

Results in Reflectance Measurement Relative to the Reference used
Various Specular Reflectance Accessories

Variable Angle Specular Reflectance Accessory (VASRA)

20-70 deg (0.5 deg steps)

Fixed Angle Accessory

Near Normal
15 degrees
30 degrees
45 degrees

Absolute Specular Reflectance Accessory (V-W design)
Cary 7000 UMS and UMA

Universal Measurement Accessory
Cary 7000 UMS and UMA
Cary 7000 UMS and UMA

Cary 7000 Universal Measurement Spectrophotometer
Overview
A turn-key, automated, UV-Vis-NIR spectrophotometer with Universal Measurement Accessory provides exceptional value, ease of use and performance – unmatched individually, unbeatable in a package.

Application Focus (Solids)
- Photonics/Optics/Thin Films
- Material research
- Semiconductor
- Military/Defense
- Chemical/Industrial
UV-Vis-NIR Spectrophotometry

Research

QA/QC Testing

Trouble Shooting

%T and %R
Angular Control
Flexibility

Cost per analysis
Automation
Productivity

Consistency
(%T and %R)
Accuracy
Performance

Easy of Use
Accessible
Reliability

Accuracy
Performance

Research

QA/QC Testing

Trouble Shooting

Agilent Technologies
Cary UMS Schematic

**Productivity**
Automated *independent* control of polarization (s or p) detector (D) position and sample rotation.

*One baseline* is needed for all %R and %T measurements, at all angles for a given polarization – dramatically reducing total collect time.

Perform all %R, %T measurements on a single system eliminating accessory change over, or reconfiguration time.

**Performance**
Absolute reflection and transmission by definition – the only difference between baseline and measurement is the sample itself.

Incident light is fixed in shape, and position, at the sample ensuring %T and %R are collected from the *same point on the sample*.

The detector has a pure line of sight of the sample. This unique *Direct View* provides the highest signal-to-noise, improving accuracy, reproducibility and productivity.
## Sandwich Detector

<table>
<thead>
<tr>
<th>Feature</th>
<th>Advantage</th>
<th>Benefit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si/InGaAs Sandwich Detector</td>
<td>Combined UV-Vis and NIR detector. No moving parts, i.e. no beam shift at detector changeover.</td>
<td>Consistent data. Large wavelength range. (UV-Vis-NIR)</td>
</tr>
</tbody>
</table>
### Direct Detector Illumination

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<tr>
<td>Direct Detector Illumination</td>
<td>All reflective, high efficiency, aluminum optics maintain high signal level and signal quality from source to detector</td>
<td>Productivity – saves time</td>
</tr>
<tr>
<td></td>
<td><strong>Alternative</strong> is using small DRAs and/or fiber optic/light pipes.</td>
<td>Loss in sensitivity, worse spectra quality or longer acquisition times.</td>
</tr>
</tbody>
</table>
Unique Wire Grid Polarizers

The Agilent Cary UV-Vis-NIR **automated polarizer** provides the next-generation of polarization control: A nano-wire grid which is lithographically laid onto a highly transmissive quartz substrate.
## Wire Grid Polarizer

<table>
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<tr>
<th>Feature</th>
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<th>Benefit</th>
</tr>
</thead>
<tbody>
<tr>
<td>High contrast ratio</td>
<td>High quality and control of S and P polarized light. better throughput</td>
<td>Superior Data Quality</td>
</tr>
<tr>
<td>Wide acceptance angle</td>
<td>Polarization of full spectrophotometer beam without compromise</td>
<td>Improving throughput leading to improved system sensitivity.</td>
</tr>
<tr>
<td>Very thin only 3 mm</td>
<td>Is smaller and more compact than Glan-Taylor/Thompson</td>
<td>extra room in the sample compartment to improve ease of analysis.</td>
</tr>
<tr>
<td>Wide wavelength range</td>
<td>250 nm – 2500 nm</td>
<td>No wavelength limitation during measurements</td>
</tr>
</tbody>
</table>
Measure with 0.02° angular precision

<table>
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</tr>
</thead>
<tbody>
<tr>
<td>High Resolution Optical Encoder</td>
<td>Fine angular control of sample rotation and detector position to 0.02 deg.</td>
<td>Robustness/Reliability - system never misses a step and permits precise sample characterization</td>
</tr>
</tbody>
</table>
P/N: G6874A, UMA Options

Edge Mount Sample Holder

Adjustable Samples Sizes: From approx 1" (25.4 mm) to 6" (150 mm) in diameter.

Thickness: Approx 200 μm to 5 mm thick.

Non contact with front/back face of sample.

Max angular range is slightly limited due to “U” shaped frame around sample (sample diameter dependent).

Please note this picture is not anodized black yet.
P/N: G6874A, UMA Options

Round Sample Holder

Sample Sizes:

1” and 2” round
(2 mm selvage area)

Dedicated, simple, easy to use. Avoids contact with center of sample.

Ensure sample is not “offset” by a thick frame around optic, or that the frame increases the actual diameter of the sample. Samples sit in recessed rim of holder.
Cube Beam Splitter Holder
Solids Autosampler

24/32 Multi Sample Holder

Rotatable and translatable (x-phi) sample holder that mounts on the UMA rotational stage

Allows rotation and translation of sample(s) for both T and R measurements at angle – multi-sample or “mapping”

Spatial resolution dictated by beam patch size at sample; 5x5 mm to 2x2 mm

8” Wafer/Large Sample Holder

Potential 300% productivity increase!
Reflectance Measurement on UMA
Transmission Measurement on UMA
Absorptance Measurement on UMA
Advanced Measurement Capabilities
Absolute Specular Reflection

Summary
Measurement of absolute specular reflectance of a SRM traceable to a NIST standard.

Results
In this figure the measured and certified spectra have been are overlaid.

Comparisons between measured value and certified value can be seen to correspond very closely across the wavelength range 250 nm – 2500 nm. Data collect was collected in ~2 min scan.
Absolute Specular Reflection

**Application**
Coating characterization and design validation. Comprehensive angular, and wavelength range, analysis of a coated silicon substrate, 200 mm diameter, 800 μm thickness.

**Challenge**
Efficient and accurate thin film design measurement by multi-angle, UV-Vis-NR spectroscopy and 2D contour plot visualization tools.
Thin Film/Coatings, Specular Reflectance

Absolute specular reflectance measured of a coated silicon substrate in the UV-Vis-NIR from near normal angles of incidence (AOI) to high grazing angles. Spectra with AOI from 6 deg to 86 deg in 1 deg increments are shown for p-polarized light. The entire spectral collect was executed in a single unattended operation.
A 2D contour plot of the previous slide helps visualize the coating dependence with AoI and wavelength and aids with locating reflection minima and maxima, e.g., minimum reflection can easily identified at 1500 nm with 70 deg AoI.
Example of ar Sample
ar Coating Near Normal Expanded
Optics – Cube Beamsplitters

Application Example
Polarizing CBS use in interferometer of nano-positioning system. Designed for HeNe laser @ 632.8 nm. Proprietary beamsplitter & anti-reflection coating made from TiO₂ & SiO₂. The two prisms are bonded with optical adhesive.

The Challenge
Measure T and R for s- and p-polarized incident without moving sample and therefore light incident on the sample. On the UMS this is easy…. AND FAST!
Optics – Cube Beamsplitters

Results

S-polarized spectra (left) desire High $R_s$ and Low $T_s$.

P-polarized spectra (right) desire High $T_p$ and Low $R_p$.

Performance is easily determined. Results can be fed back into CBS design.
RGB Cube Prism
RGB Cube Prism
RGB Cube Prism

[Graph showing the reflectance percentage (%R) as a function of wavelength (nm) with a peak at around 450 nm and a sharp drop at 500 nm, labeled as 'Detector -90 S-Polarization.']
RGB Cube Prism

Detector 180 deg
S-polarization

% T

Wavelength (nm)

0 20 40 60 80 100

400 500 600 700 800 900
RGB Cube Prism Overlay of Data
RGB Cube Prism Effect of Polarization

Detector 90 deg
Red  P-polarization
Blue  S-polarization

%R

Wavelength (nm)
400 500 600 700 800 900
Measuring Grating Efficiency
Orientation of Sample

Incident Beam

Detector

Grating Angle

10°

Diffracted Beam

Blaze Arrow

This arrow may be on the top or the bottom of the grating, but for this geometry, it must point to the left.
GRATING RESULTS

Samples Run :

300 lines per mm
1200 lines per mm
1800 lines per mm
3600 lines per mm

Data Collected at a Data Interval of 1 nm and 10 nm
Data Collected with SBW of 2 nm and 5 nm
Grating Sample on Cube Sample Holder

300 line per mm Grating on Sample Holder
Grating Sample in UMA

Detector Held at 10 degrees

Sample Stage Rotated to Calculated Angles for Grating and Wavelength which have been read from a csv file
1200 lines per mm Sample
Data Interval 1 nm SBW 2 nm
1200 lines per mm Grating

Data Interval 1 nm SBW 2 nm

P-Polarization
1200 lines per mm Grating

Data Interval 1 nm SBW 2 nm

S-Polarization
1200 lines per mm Grating

Overlay of S&P Polarization

Data Interval 1 nm SBW 2 nm
1200 lines per mm Grating

Overlay of S, P, and Average Polarization

Data Interval 1 nm SBW 2 nm
Elapsed Time for Data Collection for 1200 lines per mm Sample

1200 lines per mm P-Polarization
Start Time  5:46:57 PM
End Time  7:56:59 PM
Elapsed Time =  2 hrs 10 min 02 sec

1200 lines per mm S-Polarization
Start Time  10:44:27 PM
End Time  12:55:53 AM
Elapsed Time =  2 hrs 11 min 28 sec

Total Elapsed Time =  4 hrs 21 min 30 sec

Number of data points per scan = 1251
Comparison of Data Interval of 1 nm and 10 nm
Sample 1200 lines per mm
Overlay of P-Polarization Data Interval of 1 nm and 10 nm

Red: data interval = 1 nm  Black: data interval = 10 nm
1200 lines per mm Grating

Overlay of S-Polarization Data Interval of 1 nm and 10 nm

Red data interval = 1 nm  Black data interval = 10 nm
Scattered Reflection

Lambertian - PTFE
UMA vs DRA

DRA detects scattered light in all directions

UMA detects scattered light at one position with a cone angle of $5^\circ$
Diffuse Colour Standards
BLUE PTFE COLOUR STANDARD
UMA, Internal DRA, External DRA

All Accessories:
- Double Beam Auto Select
- UV/Vis
  - Data Interval = 1 nm
  - SBW = 4 nm
- NIR
  - Data Interval = 1 nm
  - Energy = 3

UMA:
- SAT = 0.5 s

Internal DRA
- SAT = 0.1 s

External DRA
- SAT = 0.1 s
The End
Thank You for Your Patience

Questions?

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